*भारतीय मानक*
*Indian Standard*

**IS 2630 : 2021**

# नाइट्रोबेंज़ीन — विशिष्टि

*( तीसरा पुनरीक्षण )*

# Nitrobenzene — Specification

*( Third Revision )*

ICS 70.080.30

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**April 2021** **Price Group 4**

Dye Intermediates Sectional Committee, PCD 26

## FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1964 and subsequently revised in 1973 and 1992. The standard was first revised in 1973 to incorporate a re-rectified grade of nitrobenzene in addition to the technical grade and an additional requirement for dinitrobenzene content. Second revision was undertaken to unify the requirement of the two grades of the material and incorporate the requirement of assay. GLC method has also been introduced for assay and impurities.

In this revision, method of determination of purity of nitrobenzene as well as low boiler and high boiler impurities has been incorporated. The cross-referred standards have also been updated.

Nitrobenzene is one of the most important raw materials for the dye industry, and most of it is used directly or indirectly (as aniline) in dye manufacture. It is used for the preparation of intermediate chemicals for azo dyes, including benzidine, metanilic acid, *m*-dinitrobenzene and *m*-nitrochlorobenzene, and also in the manufacture of a variety of organic substances like induline quinoline, magenta and nigrosine. Among its numerous applications as a solvent, it is used in the petroleum industry as a selective solvent. It occasionally serves as a mild oxidizing agent in organic reactions, such as the fuchsin fusion and the quinoline synthesis.

The composition of the Committee, responsible for the formulation of this standard is given at Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values ( *revised* )’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard*

# NITROBENZENE — SPECIFICATION

*( Third Revision )*

## 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for nitrobenzene.

## 2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute the provisions of the standards. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

| *IS No.* | *Title* |
|---|---|
| 1070 : 1992 | Reagent grade water specification (*third revision*) |
| 1260 (Part 1) : 1973 | Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*) |
| 1446 : 2002 | Classification of dangerous goods (*second revision*) |
| 2362 : 1993 | Determination of water by the karl fischer method — Test method (*second revision*) |
| 5299 : 2001 | Method of sampling and test for dye intermediates (*first revision)* |

## 3 REQUIREMENTS

### 3.1 Description

The material shall be a pale to dark yellow liquid, free from visible impurities and water.

**3.2** The material shall also comply with the requirements given in Table 1.

## 4 PACKING AND MARKING

### 4.1 Packing

**4.1.1** The material shall be packed securely in closed opaque containers or as agreed between the buyer and the seller, protected from light and shall be stored in cool place.

**Table 1 Requirements for Nitrobenzene**

( *Clauses* 3.2 *and* 6.1 )

| **Sl No.** | **Characteristic** | **Requirements** | **Method of Test, Ref. To** | |
|---|---|---|---|---|
| | | | IS | Annex |
| (1) | (2) | (3) | (4) | (5) |
| i) | Relative density at 27 °C/27 °C | 1.197 to 1.20 | IS 5299 | – |
| ii) | Distillation range at 760 mm Hg. | 2 to 97 ml shall Distil between 209 to 212 °C | IS 5299 | – |
| iii) | Crystallization point °C, *Min* | 5.4 | IS 5299 | – |
| iv) | Moisture, percent by Mass, *Max* | 0.2 | IS 2362 | – |
| v) | *p*H, *Min* | 5.5 | – | Annex A |
| vi) | Dinitrobenzene, percent by Mass, *Max* | 0.1 | – | Annex B |
| vii) | Assay, percent by Mass, *Max* | 99.5 | – | Annex C or D |
| viii) | Low boilers (Benzene) percent by Mass, *Max* | 0.2 | | |
| ix) | High boilers (*o*-nitrotoluene, *p*-nitrotoluene, *p*-nitrochlorobenzene, and *m*-dinitrobenzene), percent by mass, *Max* | 0.2 | | |

**4.1.2** All the containers in which the material is packed shall be dry, clean, free from substances soluble in nitrobenzene and leakproof.

**4.1.3** Necessary safeguards against the risk arising from the storage and handling of large volumes of poisonous liquids (*see* IS 1446) shall be provided and due precautions shall be taken [*see* IS 1260 (Part 1)].

**4.1.4** Except when opened for the purpose for cleaning and rendering them free from nitrobenzene vapour, all empty tanks or other containers shall be kept securely closed, provided they have been leaned and free from nitrobenzene.

### 4.2 Marking

**4.2.1** The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

**4.2.2** Each container shall be marked with the following.

a) Name of material;
b) Indication of the source of material;
c) Net mass of the material;
d) Month and year of manufacture;
e) Batch number, if any; and
f) The minimum cautionary notice worded as under:
   "POISON, LIQUID AND VAPOUR HAZARDOUS, RAPIDLY ABSORBED THROUGHSKIN"

**4.2.3** *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## 5 SAMPLING

**5.1** Representative sample shall be drawn as per prescribed in **3** of IS 5299.

**5.2** For bulk storage tanks, representative sample will be drawn from pump discharge after thorough circulation of the material in tank.

## 6 TEST METHOD

**6.1** Test shall be carried our according to the methods prescribed in Annexes and specifications indicated in col 4 of Table 1.

### 6.2 Quality or Reagent

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

# ANNEX A

[ *Table* 1, *Sl No.* (v) ]

## DETERMINATION OF *p*H

**A-1 APPARATUS** — *p*H meter.

**A-2 ELECTRODE** — Glass electrode.

**A-3 REAGENT** — Buffer solution, *p*H-4, *p*H-7.0 and *p*H-9.

### A-4 PROCEDURE OF CALIBRATION OF *p*H METER

Clean the electrode with distilled water (*see* IS 1070) and put into standard buffer solution as per requirement. Check the reading on display and calibrate the *p*H meter with respect to value of buffer solution.

### A-5 PROCEDURE

Take 100 g of sample in a conical flask with stopper and add 100 ml of boiling water into it. After closing the flask with stopper, shake it vigorously for one minute. Cool it quickly with cold water (take off the stopper). Transfer the supernatant liquid into a beaker and measure its *p*H. Note down the direct reading displayed on the *p*H meter.

# ANNEX B

[ *Table* 1, *Sl No.* (vi) ]

## DETERMINATION OF DINITROBENZENE CONTENT

### B-1 REAGENTS

**B-1.1 Acetone,** free from alcohol and aldehydes.

**B-1.2 Potassium Hydroxide Solution,** 10 percent (m/v).

**B-1.3 *m*-dinitrobenzene**, crystalline, melting point: 89.7 °C.

### B-2 PROCEDURE

**B-2.1** Weigh accurately about 0.5 g of sample using a weighing bottle and transfer to 100 ml measuring flask. Add 70 ml acetone, stir and add 4 ml of potassium hydroxide solution. Stir vigorously and make up to 100 ml with acetone.

**B-2.1.1** Prepare several solutions containing 1, 2, 2.5, 3 µg and so on of *m*-dinitrobenzene in 1 ml of acetone containing 4 ml of potassium hydroxide solution. Measure absorbance after 5 minutes at 565 nm and plot a calibration curve of absorbance versus µg per ml of *m*-dinitrobenzene in acetone.

**B-2.1.2** Measure absorbance of the test solution at 565 run and determine its *m*-dinitrobenzene content in µg/ml from the calibration curve.

### B-3 CALCULATION

*m*-dinitrobenzene content, percent by mass = $m_1/M \times 100$

where

$m_1$ = micrograms of m-dinitrobenzene per ml read from calibration curve, and

$M$ = mass in g, of sample present in 100 ml of test solution.

# ANNEX C

[ *Table* 1, *Sl No.* (vii), (viii) *and* (ix) ]

## DETERMINATION OF ASSAY AND IMPURITIES

### C-1 OUTLINE OF THE METHOD

The contents of nitrobenzene and probable impurities like benzene, ortho, meta and para nitrotoluene and dinitrobenzene are determined by gas liquid chromatographic method. A sample of the material injected into the gas chromatograph column is carried by carrier gas and during its movement the constituents undergo distribution at different rates. The separated constituents emerge from the other end of the column which are detected by suitable means where response is related to the amount of a specific component leaving the column.

## C-2 APPARATUS

### C-2.1 Gas Chromatograph

A suitable gas chromatograph with flame ionization detector and temperature programmer and following details:

| | | |
|---|---|---|
| Column | : | Dimension — length: 2000 mm, Internal diameter: 2 mm, Stationary phase: packing with 2.5 percent OV17 + 3.66 percent QFI on Chromosorb WHP 80/100 mesh. |
| Data acquisition system | : | Any suitable data integrator or PC based GC software which can handle features like external/internal standard calculations, peak grouping etc. |
| Carrier gas | : | Nitrogen, flow rate 30 ml/min<br>Air, flow rate 300 ml/min<br>Hydrogen, flow rate 30 ml/min |
| Temperature | : | Column temperature 95 °C for 17 min, programmed 15 °C/min, heating rate to 170 °C retained for 10 minutes. |
| Injection volume | : | 0.2 µl |

## C-3 REAGENTS

**C-3.1 Benzene**, A. R. grade.

**C-3.2 Nitrobenzene**, A. R. grade.

**C-3.3 *o*-Nitrotoluene**, A. R. grade.

**C-3.4 *p*-Nitrotoluene**, A. R. grade.

**C-3.5 *m*-Dinitrobenzene**, A. R. grade.

**C-3.6 *p*-Nitrochlorobenzene**, A. R. grade (as an internal standard).

## C-4 PROCEDURE

### C-4.1 Calibration Factor

Prepare a calibration mixture of known components for which the calibration factor is to be determined together with the internal standard. The calibration factor is calculated by the following formula:

$$K_n = \frac{X_n \times A_s}{X_s \times A_n}$$

where

$K_n$ = calibration factor for component n,
$X_n$ = mass percent of component n in the sample,
$A_s$ = peak area of internal standards,
$X_s$ = mass percent of internal standard s in the sample, and
$A_n$ = peak area of component n.

### C-4.2 Interpretation of Chromatogram

Elution order of components is as follows:

a) benzene (low boiler);
b) nitrobenzene;
c) *o*-nitrotoluene (high boiler);
d) *p*-nitrotoluene (high boiler);
e) *p*-nitrochlorobenzene (high boiler), an internal standard; and
f) *m*-nitrobenzene (high boiler).

## C-5 CALCULATION

Component n in the sample percent by mass =

$$\frac{K_n \times A_n \times X_s}{A_s}$$

where

$K_n$ = calibration factor;
$A_n$ = peak area for component n;
$X_s$ = mass present of internal standard added to the sample; and
$A_s$ = peak area for internal standard.

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, $H_2$, $N_2$) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

# ANNEX D

[ *Table* 1, *Sl No*. (vii), (viii) *and* (ix) ]

## DETERMINATION OF ASSAY AND IMPURITIES

### D-1 SCOPE

This method is used for the determination of purity of nitrobenzene as well as low boiler and high boiler impurities.

### D-2 OUTLINE OF THE METHOD

The contents of nitrobenzene and probable impurities like benzene, ortho, meta and para nitrotoluene and dinitrobenzene are determined by gas liquid chromatographic method. A sample of the material injected into the gas chromatograph column is carried by carrier gas and during its movement the constituents undergo distribution at different rates. The separated constituents emerge from the other end of the column which are detected by suitable means where response is related to the amount of a specific component leaving the column.

### D-3 APPARATUS

**D-3.1 Gas Chromatograph**

A suitable gas chromatograph with flame ionization detector and temperature programmer and following details:

| | | |
|---|---|---|
| Column | : | Dimension: length: 30 meters, internal diameter: 0.32 mm, film thickness: 1.0 µm, stationary phase having fused silica with combination of polar and non-polar phase like, 85-90 percent dimethylpolysiloxane and 10-15 percent polyethylene glycol or cyanopropylphenyl. Columns like DX-1, BP-10, DB1701, RTX 1701 etc are suitable for this. |
| Data acquisition system | : | Any suitable data integrator or PC based GC software which can handle features like external/internal standard calculations, peak grouping etc. |
| Carrier gas | : | Hydrogen |
| | | Pressure at column head: 15 psi |
| | | Hydrogen: flow rate 30 ml/min |
| | | Split flow: 200 ml/min |
| | | Air: flow rate 300 ml/min |
| Temperature | : | Column temperature 120 °C for 5 min, programmed 15 °C/min, heating rate to 160 °C retained for 4.5 min |
| Detector | : | FID |
| | | Detector temperature: 240 °C |
| | | Injector temperature: 240 °C |
| Injection volume | : | 0.5 µl |

### D-4 REAGENTS

**D-4.1 Benzene**, A. R. grade.

**D-4.2 Nitrobenzene**, A. R. grade.

**D-4.3 *o*-Nitrotoluene**, A. R. grade.

**D-4.4 *p*-Nitrotoluene**, A. R. grade.

**D-4.5 *m*-Nitrotoluene**, A. R. grade.

**D-4.6 *m*-Dinitrobenzene**, A. R. grade.

### D-5 PROCEDURE

**D-5.1 Calibration Factor**

Prepare a calibration mixture of known components for which the calibration factor is to be determined. The calibration factor is calculated by the following formula:

$$\text{Response Factor (RF)} = A_1/C_1$$

where

$A_1$ = peak area of nitrobenzene in calibration standard; and

$C_1$ = concentration of nitrobenzene in calibration standard

Or

Use suitable software for auto calibration with respect to mass of calibration standard.

**D-5.2 Interpretation of Chromatogram**

Elution order of components is as follows:

a) benzene (low boiler);
b) nitrobenzene;
c) *o*-nitrotoluene (high boiler);
d) *m*-nitrotoluene (high boiler);
e) *p*-nitrotoluene (high boiler); and
f) dinitrobenzene (high boiler).

## D-5 CALCULATION

Concentration of component.

$$\frac{A_2 \times C_1}{A_1} \text{ or } = \frac{A_2}{RF}$$

where

$A_1$ = peak area of nitrobenzene of calibration standard;

$C_1$ = concentration of nitrobenzene in calibration standard; and

$A_2$ = peak area of nitrobenzene of sample.

**D-5.1 Report**

Report the concentration of each peak by normalizing the amount of all peaks or use suitable software for auto calculation with respect to calibration standard.

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, $H_2$, $N_2$) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

# ANNEX E

( *Foreword* )

## COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

| *Organization* | *Representative(s)* |
|---|---|
| ICT, Mumbai | Prof P. M. Bhate (***Chairman***) |
| Aarti Industries Limited, Mumbai | Shri Kirit H. Desai |
| Alkyl Amines Chemicals Limited, Mumbai | Shri Kirat Patel<br>Shri S. V. Nikumbhe (*Alternate*) |
| Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat | Shri Mansukh H. Vekaria |
| Archroma, Thane | Dr Rajesh Ramamurthy<br>Shri Hemant Mhadeshwar (*Alternate*) |
| Atul Limited, Valsad, Gujarat | Dr J. G. Desai<br>Dr M. U. Rahman (*Alternate*) |
| BASF India Limited, Mumbai | Shri Uday Kulkarni |
| Central Revenue Control Laboratory (CRCL), New Delhi | Dr T. A. Sreenivasa Rao<br>Shri Praful Dalal (*Alternate*) |
| Colourtex Industries Limited, Mumbai | Dr Pankaj Desai<br>Shri Ismail Habibulla (*Alternate*) |
| Deepak Nitrite Limited, Vadodara | Shri Sailash Raval<br>Dr Mangalya Kar (*Alternate* I)<br>Dr J. K. Astik (*Alternate* II) |
| Ecological and Toxicological Association of Dyes (ETAD), Vadodra | Dr Pariti Siva Rama Kumar |
| Gujarat Dyestuffs Manufacturers Association (GDMA), Ahmedabad | Shri R. S. Patel<br>Shri Bansibhai Patal (*Alternate*) |
| Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad | Shri Y. A. Tai |
| Heubach Colour Private Limited, Mumbai | Shri J. I. Sevak<br>Shri Vinod Madhukar (*Alternate*) |
| Indian Chemical Council, Mumbai | Shri P. S. Singh |
| Jay Chemical Industries, Ahmedabad | Shri Vilpesh Yadav<br>Shrimati Maitri Vyas (*Alternate*) |
| Department of Chemicals & Petrochemicals, Ministry of Chemicals & Fertilizers, New Delhi | Shri Jasbir Singh |
| Ministry of Defence (Research and Development), New Delhi | Dr Vijay Tak |
| National Test House (er), Kolkata | Shri P. K. Chakraborty<br>Shri Y. C. Nijhawan (*Alternate*) |

| *Organization* | *Representative(s)* |
|---|---|
| Sudarshan Chemical Industries Limited, Pune | Dr Jaychandran M. Nair |
| The Bombay Textile Research Association | Dr Ashok N. Desai |
| The Dyestuffs Manufacturers Association of India (DMAI), Mumbai | Shri V. R. Kanetkar |
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Shri Vijay Kumar Gupta
Scientist 'D' (PCD), BIS

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## Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
|---|---|---|
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